



 **COPY**

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No. :10/659,090

Confirmation No.:2724

Applicant :Christopher J. Nagel

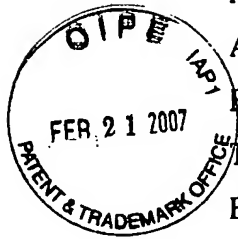
Filed :September 10, 2003

TC/A.U. :1751

Examiner :Mark T. Kopec

Docket No. :2751.2001 US2

Title: COMPOSITION OF MATTER TAILORING: SYSTEM I



<b>CERTIFICATE OF MAILING OR TRANSMISSION</b>	
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Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

**DECLARATION UNDER 37 CFR 1.132**

Sir:

I, Christopher J. Nagel, of 28 Highland Circle, Wayland MA 01778, am the sole inventor of the above identified application. I am the sole inventor of the above identified patent application.

I am attaching a detailed description of each of the analytical techniques described in this declaration and in my Declaration under 37 CFR 1.132 filed on March 23, 2006. These techniques were performed by third party companies to confirm the change in matter achieved through tailoring (Exhibit 1). GDMS was obtained from SHIVA Technologies of Syracuse, New York; XRF was obtained from the University of Western Ontario, London, Ontario; PIXE was obtained from Elemental Analysis Incorporated,

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Lexington, Kentucky; and GDOES was obtained from Twin Analytical of Independence, Ohio.

I have been asked to clarify the process of manufacture for the copper samples described in my earlier Declaration under 37 CFR 1.132. The copper sample in the declaration, as are the samples described herein, refer to the ingot number of the sample. Copper 14-00-01 corresponds to Example 11 of the present application.

As explained previously, third party data confirms that the manufactured copper ingot contains a different elemental signature from naturally occurring copper (Exhibit 3) and my earlier Declaration. To show that the process of the present invention is not unique to copper, I am also attaching third party data for the following elements: aluminum (Exhibit 4), silicon (Exhibit 5), iron (Exhibit 6), nickel (Exhibit 7), and cobalt (Exhibit 8) together with their respective experimental procedures. Please note that the aluminum example is not described in the current application, but rather in my copending application number 11/063,694. The aluminum ingot was produced in accordance with the methods described in these patent applications. The third party data confirmed the unique electronic characteristic of each tailored system. Please note that the spreadsheets in Exhibits 3-8 include GD-MS analyses of each of the starting materials. Further Exhibits 3-8 include the manufacturer's specifications of the starting materials for each system.

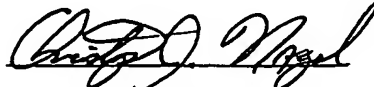
I have been asked to provide visual support for the teachings in the specification of the magnetic properties of the tailored copper produced by the present process. I am attaching six photographs of the tailored copper ingot 14-00-01, corresponding to Example 11 of the present specification, (Exhibit 2). I personally observed the magnetic properties described in the slides of Exhibit 2.

I have been asked to analyze a "magnetic copper" sample obtained from Goodfellow Cambridge Limited. As shown in Exhibit 2, the photographs reveal the presence of iron impregnated in copper. In my opinion, the substantial presence of iron creates the magnetic property. Therefore, it is my opinion that the material is not copper substantially free of other metals.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further

Application No.: 10/659,090  
Declaration by Christopher J. Nagel

that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.



Christopher J. Nagel

# Natural Silicon Manufacturer's Specification



13 Foster St., P.O. Box 181, Bergenfield, NJ 07621  
TEL: (800) 486-2438 • FAX: (201) 387-0281

a Division of MICRON METALS, Inc.

## Certificate of Analysis

SOLD TO: QUANTUM CATALYTICS  
CUSTOMER PO: 20429  
SHIP DATE: 30 MAR 01  
CATALOG NUMBER [REDACTED]  
LOT #: 304007  
ITEM NAME: SILICON METAL POWDER  
CAS NUMBER: 7440-21-3  
DOCUMENT: 103215  
QUANTITY: 26 LBS

## CHEMICAL ANALYSIS

SILICON 99.99+%

## SCREEN ANALYSIS

100% +100 mesh

The above analysis is carried out as part of our internal quality control testing and is based upon our analysis methods

We do not assume any warranty, liability, or risk based on such findings. Our quality is warranted within the scope of our general sales conditions.

Very Truly Yours,

M. Gerald, QA

## **EXPERIMENTAL PROCEDURE FOR SILICON 15-01-01**

### **EXAMPLE 7**

A cylindrical alumina-based crucible (99.68%  $\text{Al}_2\text{O}_3$ , 0.07%  $\text{SiO}_2$ , 0.08%  $\text{Fe}_2\text{O}_3$ , 0.04%  $\text{CaO}$ , 0.12%  $\text{Na}_2\text{O}_3$ ; 4.5" O.D. x 3.75" I.D. x 10" depth) of a 100 pound induction furnace reactor (Inductotherm) fitted with a 75-30R Powertrak power supply was charged with 700 g Silicon (100.00% purity), through its charging port. The reactor was fitted with a graphite cap and a ceramic liner (i.e., the crucible, from Engineering Ceramics). During the entire procedure, a slight positive pressure of nitrogen (~0.5 psi) was maintained in the reactor using a continuous backspace purge. The reactor was heated to the metal charge liquidus point plus 300°F, at a rate no greater than 300°F/hour as limited by the integrity of the crucible. The induction furnace operated in the frequency range of 0 kHz to 3000kHz, with frequency determined by a temperature-controlled feedback loop implementing an Omega Model CN 300 temperature controller. Upon reaching 2800°F, the reactor was charged with an additional 400 g Silicon again using a rate no greater than 300°F/hour.

The temperature was again increased to 2900°F again using a rate no greater than 300°F/hour. When this temperature was reached, graphite saturation assemblies (3/8" OD, 36" long high purity (5 ppm impurities) graphite rods) were inserted to the bottom of the Silicon charge through ports located in the top plate. The Silicon was held at 2900°F for 4 hours. Every 30 minutes during the hold period, an attempt was made to lower the graphite saturation assemblies as dissolution occurred. As the Silicon became saturated with carbon, the graphite saturation assemblies were consumed. After the 4 hour hold period was complete, the graphite saturation assemblies were removed.

The reactor temperature was increased to 2976°F over 7 minutes. The temperature was then varied between 2920°F and 2976°F for 15 cycles. Each cycle consisted of lowering the temperature continuously over 7 minutes and raising the temperature continuously over 7 minutes. After the 15 cycles were completed, a gas addition lance was lowered into the molten metal to a position approximately 2" from the bottom of the reactor and a 0.15 L/min flow of argon was begun. The temperature of the Silicon was varied over another 5 cycles between 2920°F and 2976°F.

After the fifth cycle, the reactor temperature was lowered to 2900°F over a 10 minute period with continued argon addition. The graphite saturation assemblies were reinstalled in the Silicon and remained there for 1 hour. The graphite saturation assemblies were removed.

The reactor temperature was lowered to 2895°F over 5 minutes. The reactor was held at this temperature for 5 minutes with continued argon addition. The temperature was then varied between 2886°F and 2895°F over 20 cycles. Each cycle consisted of lowering the temperature continuously over 9 minutes and raising the temperature continuously over 9 minutes. The argon addition ceased after completion of the 20 cycles.

The reactor temperature was lowered to 2873°F over 5 minutes. The temperature was varied between 2868°F and 2873°F over 4 ½ cycles. Each cycle consisted of lowering the temperature continuously over 5 minutes and raising the temperature continuously over 3 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added.

The reactor temperature was lowered to 2863°F over 5 minutes. The temperature was varied between 2811°F and 2863°F for 15.5 cycles. Each cycle consisted of lowering the temperature continuously over 15 minutes and raising the temperature continuously over 15 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added. All gas addition, except for the purge of nitrogen ceased after the 15.5 cycles were completed.

The temperature was varied between 2833°F and 2811°F for one cycle. The cycle consisted of raising the temperature continuously over 15 minutes and lowering the temperature continuously over 15 minutes. The gas addition lance was removed.

The reactor temperature was slowly cooled by lowering the induction furnace power to 1 kW or less as the ingot cooled. After solidification, the Silicon was cooled to approximately ambient temperature in water.

## ANALYTICAL PROTOCOLS

XRF, grain size, magnetism, and chemical reactivity measurements were carried out by the procedures described in Example 1.

## ANALYTICAL RESULTS

An x-ray fluorescence analysis of the silicon sample is provided in Figures 45A and 45B, with the  $K_{\alpha}$  and  $L_{\alpha}$  peaks of a silicon control standard shown for reference.

An x-ray fluorescence analysis of the silicon sample is provided in Figure 46A, with the  $K_{\alpha}$  peak of an aluminum control standard shown for reference.

An x-ray fluorescence analysis of the silicon sample is provided in Figure 46B, with the  $K_{\alpha}$  peak of a titanium control standard shown for reference.

An x-ray fluorescence analysis of the silicon sample is provided in Figure 47A, with the  $K_{\alpha}$  peak of a sulfur control standard shown for reference.

An x-ray fluorescence analysis of the silicon sample is provided in Figure 47B, with the  $K_{\alpha}$  peak of a chlorine (from potassium chloride) control standard shown for reference.

An x-ray fluorescence analysis of the silicon sample is provided in Figure 48A, with the  $K_{\alpha}$  peak of a gallium control standard shown for reference.

An x-ray fluorescence analysis of the silicon sample is provided in Figure 48B, with the  $K_{\alpha}$  peak of a potassium control standard shown for reference.

Summary data showing the apparent elemental composition of the product of Example 7 is shown in Tables 24-27, as was measured by an XRF analysis using a Uniquant software package. The apparent elemental composition of the product varies by position, which is indicated in each table.

No unexpected magnetic activity or chemical reactivity were recorded for the ingot. The manufactured silicon system did appear shiny on its axial (top) face and dull on its radial (side) face. The ingot retained minimal refractory upon removal from the reactor.

# Natural Copper Manufacturer's Specification



**Hickman Williams & Company**

MATERIALS FOR THE METALS INDUSTRY  
CHICAGO • CINCINNATI • CLEVELAND  
DETROIT • PHILADELPHIA • PITTSBURGH  
CAMBRIDGE, ONTARIO

## CERTIFICATE OF ANALYSIS

TO: Quantum Catalytics

DATE: 11/10/99

LOT # 120

PO # 10190

MATERIAL: COPPER CHOPS

WEIGHT: 5000 lbs.

TIN	.0014	COPPER	99.98
IRON	.0037	SELENIUM	ND
ANTIMONY	ND	ARSENIC	ND
SULFUR	.0007	BISMUTH	ND
CADMIUM	ND	COBALT	ND
ZINC	ND	NICKEL	.0008
SILVER	ND	LEAD	.0038
PHOSPHOROUS	ND	MANGANESE	ND

\*less than

Certified by: *Russell L. Echerington*

Tin • Aluminum • Copper • Zinc • Lead • Antimony • Magnesium • Cadmium • Brass • Bronze



## **EXPERIMENTAL PROCEDURE FOR COPPER 14-00-01**

### **(EXAMPLE 11)**

A cylindrical alumina-based crucible (89.07%  $\text{Al}_2\text{O}_3$ , 10.37%  $\text{SiO}_2$ , 0.16%  $\text{TiO}_2$ , 0.15%  $\text{Fe}_2\text{O}_3$ , 0.03%  $\text{CaO}$ , 0.01%  $\text{MgO}$ , 0.02%  $\text{Na}_2\text{O}_3$ , 0.02%  $\text{K}_2\text{O}$ ; 9" O.D. x 7.75" I.D. x 14" depth) of a 100 pound induction furnace reactor supplied by Inductotherm, fitted with a 75-30R Powertrak power supply, was charged with 100 pounds copper (99.98% purity) through its charging port. During the entire procedure, a slight positive pressure of nitrogen (~0.5 psi) was maintained in the reactor using a continuous backspace purge. The reactor was heated to the metal charge liquidus point plus 300°F, at a rate no greater than 300°F/hour, as limited by the integrity of the crucible. The induction furnace operated in the frequency range of 0 kHz to 3000 kHz, with frequency determined by a temperature-controlled feedback loop implementing an Omega Model CN300 temperature controller.

The temperature was again increased to 2462°F again using a rate no greater than 300°F/hour. When this temperature was reached, graphite saturation assemblies (3/8" OD, 36" long high purity (<5 ppm impurities) graphite rods) were inserted to the bottom of the copper charge through ports located in the top plate. The copper was held at 2462°F for 4 hours. Every 30 minutes during the hold period, an attempt was made to lower the graphite saturation assemblies as dissolution occurred. As the copper became saturated with carbon, the graphite saturation assemblies were consumed. After the 4 hour hold period was complete, the graphite saturation assemblies were removed.

The reactor temperature was increased to 2480°F over 7 minutes. The temperature was then varied between 2480°F and 2530°F for 15 cycles. Each cycle consisted of raising the temperature continuously over 7 minutes and lowering the temperature continuously over 7 minutes. After the 15 cycles were completed, a gas addition lance was lowered into the molten metal to a position approximately 2" from the bottom of the reactor and a 1.5 L/min flow of argon was begun. The temperature of the copper was varied over another 5 cycles between 2480°F and 2530°F.

After the fifth cycle, the reactor temperature was lowered to 2462°F over a 10 minute period with continued argon addition. The graphite saturation assemblies were

reinstalled in the copper and remained there for 1 hour. The graphite saturation assemblies were removed.

The reactor temperature was lowered to 2459°F over 5 minutes. The reactor was held at this temperature for 5 minutes with continued argon addition. The temperature was then varied between 2459°F and 2453°F over 20 cycles. Each cycle consisted of lowering the temperature continuously over 9 minutes and raising the temperature continuously over 9 minutes. The argon addition ceased after completion of the 20 cycles.

The reactor temperature was lowered to 2450°F over 5 minutes. The temperature was varied between 2450°F and 2441°F over 4 ½ cycles. Each cycle consisted of lowering the temperature continuously over 5 minutes and raising the temperature continuously over 3 minutes. In addition, while raising the temperature, a 1.5 L/min flow of argon was added, and while lowering the temperature, a 1.5 L/min flow of nitrogen was added.

The reactor temperature was lowered to 2438°F over 5 minutes. The temperature was varied between 2438°F and 2406°F for 15.5 cycles. Each cycle consisted of lowering the temperature continuously over 15 minutes and raising the temperature continuously over 15 minutes. In addition, while raising the temperature, a 1.5 L/min flow of argon was added, and while lowering the temperature, a 1.5 L/min flow of nitrogen was added. All gas addition, except for the purge of nitrogen ceased after the 15.5 cycles were completed.

The temperature was varied between 2406°F and 2419°F for one cycle. The cycle consisted of raising the temperature continuously over 15 minutes and lowering the temperature continuously over 15 minutes. The gas addition lance was removed.

The reactor temperature was rapidly cooled by quenching in water, so that the copper solidified into an ingot.

#### ANALYTICAL PROTOCOLS

XRF, grain size, magnetism, and chemical reactivity measurements were carried out by the procedures described in Example 1.

## ANALYTICAL RESULTS

Summary data showing the apparent elemental composition of the product of Example 11 is shown in Tables 32-33, as was measured by an XRF analysis using a Uniquant software package. The apparent elemental composition of the product varies by position, which is indicated in each table.

Immediately after the method described above was completed, multiple discrete magnetic spots attracted by a 1/8" diameter neodymium iron boron magnet were observed in a sinusoidal pattern. The ingot exhibited point attraction to iron filings at reduced temperatures at or near 77 K. Over days to months, the strength of the magnetic attraction decreased on a fraction of the locations exhibiting magnetic attraction or attraction to iron filings.

Various forms of ligated chlorine (e.g., HCl and MCl, where M is a metal as defined above) readily reacted with the manufactured copper form yielding product distributions distinguishable from natural copper, thereby demonstrating a change in chemical reactivity. This reactivity increased over time.

Extremely large grain sizes (i.e., greater than 1") were observed, which is uncharacteristic and previously unreported in natural copper systems (typically, copper grains sizes are 10-100  $\mu\text{m}$ ). Unique changes in coloration were observed with the crossing of grain boundaries; however, the overall coloration mimicked natural copper.

## **EXHIBIT 1**

### **Analytical Techniques**

## **Analytical Techniques**

This document reviews the analytical techniques used to perform the elemental analyses described herein. The descriptions (including lower detection limits, LDLs) are taken primarily from reference and textbooks<sup>1,2,3</sup> and the sources of the individual third-party analytical laboratories that performed the analysis. These sources are referenced for the reader who is interested in a more in-depth description, including schematics. The descriptions below are abbreviated extractions from each of these sources. In addition, Table 1 gives an overview of the analytical protocol followed by the investigators to obtain the submitted data. Table 2 delineates the activation and detection methods used by each method allowing a ready comparison of the similarities and differences of each technique.

### **Glow Discharge Mass Spectrometry (GD-MS)<sup>4</sup>**

*Bulk composition analysis (continuous sample consumption) based on the weights of ions sputtered from the sample using a DC plasma. Typical LDLs on the order of parts per billion (ppb, element dependent). Method identical to GD-OES except detection/identification method based on mass, not electronic character.*

- Solid sample is atomized by sputtering in a low-pressure DC plasma
- Sputtered atoms are then ionized and extracted into a mass analyzer for separation and detection
- Mass to charge ratios are well known allowing identification of elemental species

Positive ions formed in the Argon plasma glow are accelerated toward the sample. Upon impacting the surface of the sample, neutral particles (predominantly individual atoms) and positive ions are released. The sputtered neutral particles diffuse across the plasma “dark space” and are ionized through interaction with the metastable argon ions of the plasma primarily forming singly charged ions. These singly charged ions are extracted into a mass analyzer for separation and detection. The Mass to Charge ratios are used for elemental species identification.

### **Glow Discharge Optical Emission Spectrometry (GD-OES)<sup>5</sup>**

*Quantitative Depth Analysis (near surface 0.05-150 mm) based on the optical emissions (wavelength) of material sputtered from sample surface. Typical LDLs of ~5*

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<sup>1</sup>Handbook of Instrumental Techniques for Analytical Chemistry, Frank A. Settle (Ed), Prentice Hall, June 1997.

<sup>2</sup>Handbook of Analytical Techniques, Vol 1 & 2; Helmut Günzler, Alex Williams (Eds) John Wiley & Sons, Inc., May 2001

<sup>3</sup>Practical Handbook of Spectroscopy, J.W. Robinson (Ed.), CRC Press, Inc., 1991

<sup>4</sup>Shiva Technologies, Inc. Trace Elemental Analysis, Glow discharge Mass Spectrometry Principles, ©2001. <http://www.shivatec.com/new/gdmsdesc.php4>

<sup>5</sup>Twin Analytical, Glow Discharge Optical Emission Spectrometer, Instrumentation, ©2003-2005. <http://www.twinanalytical.com/instruments/GD-OES.php>

*ppm. Method identical to GD-MS except detection/identification method based on electronic structure.*

- Solid sample is atomized by sputtering in an Ar plasma
- Sputtered atoms are then ionized emitting characteristic light emissions
- The spectral lines (wavelengths) are indicative of the elemental composition

Positively charged Ar ions ( $\text{Ar}^+$ ) are accelerated, continuously bombarding the sample surface, causing atoms to be ejected. The atoms are excited or ionized in the plasma gas, emitting energy in the form of characteristic light emissions. The light emitted by the sample passes into the spectrometer to photomultiplier tubes that quantify the intensity of the spectral lines (wavelengths). Each element has its own characteristic spectrum and unique wavelength fingerprint (indicative of electron levels) allowing elemental identification.<sup>6,7</sup>

### **Particle Induced X-ray Emission Spectrometry (PIXE)<sup>8</sup>**

*Semi-bulk (40 mm penetration depth) analysis based on the emission energies characteristic of electronic orbital shifts. Typical LDLs are on the order of parts per million or higher and are dependent on the element. XRF and PIXE use identical detection methods to identify elemental components of the sample. The sample excitation methods differ in that XRF uses hard X-ray bombardment, while PIXE uses particle (in this case proton) bombardment to induce X-ray emissions.*

- Sample surface is bombarded with protons ( $\text{H}^+$ , 40mm penetration, used in subject technique) or alpha particles ( $\text{He}^{++}$ , 8 mm)
- K-shell electrons are knocked out of innermost electron shell
- Electrons from outer shells replace inner shell electrons simultaneously emitting an x-ray
- Known x-ray energy emissions allow elemental identification

Particle Induced X-ray Emission bombards samples with two types of ions, alpha particles ( $\text{He}^{++}$ ) and protons ( $\text{H}^+$ ), at high energies. These particles knock out sample electrons from the innermost electron shell, called the K-shell. To fill this vacancy, electrons from the outer shells replace the inner shell electrons with a corresponding emission in energy in the form of an x-ray. Each element has its own characteristic x-ray emission, a unique property, allowing elemental identification. Depth of penetration: 40mm ~ 60mm for copper; 60mm ~ 80mm for aluminum.

### **X-ray Fluorescence Spectrometry<sup>9</sup>**

*Semi-bulk composition analysis (1-3 mm penetration depth) based on the emission energies characteristic of electronic orbital shifts. Typical LDLs are approximately 20-*

<sup>6</sup> Glow Discharge Optical Emission Spectrometry, Richard Payling, Delwyn Jones, Arne Bengtson (Eds), John Wiley & Sons, Ltd., 1997.

<sup>7</sup> Glow Discharge Plasmas in Analytical Spectroscopy, R. Kenneth Marcus, Jose A. C. Broekaert (Eds.), John Wiley & Sons, Ltd., 2003.

<sup>8</sup> Elemental Analysis Inc., "Proton Induced X-ray Emission"; <http://www.elementalanalysis.com/pixe/>

<sup>9</sup> Eastern Illinois University, Doug Klarup, Ph.D., Chair Dept. of Chemistry, "X-ray Fluorescence Spectroscopy", <http://www.ux1.eiu.edu/~dgklarup/nsfclli/instrumentconcepts/xrf.pdf>

*50 ppm and are dependent on the individual elements present in the sample matrix. XRF and PIXE use identical detection methods to identify elemental components of the sample. The sample excitation methods differ in that XRF uses X-ray bombardment, while PIXE uses particle (in this case proton) bombardment to induce X-ray emissions.*

- Sample surface is bombarded with x-rays*
- K-shell electrons are knocked out of innermost electron shell*
- Electrons from outer shells replace inner shell electrons simultaneously emitting an x-ray*
- Known x-ray energy emissions allow elemental identification*

A quant of the primary X-ray beam excites atoms of the elements present in the sample removing electrons from the inner-most shell (K-shell). The vacancies are filled with electrons from a higher energy level, the L-shell. As the electron moves to a lower energy shell, a fluorescence quant (emitted x-ray) is created. Its energy is determined by the energetic difference between the L- and the K-shell and is thus characteristic of the element. For these analyses a sequential wavelength dispersive spectrometer (WDS) was used in which specific emission lines are used to determine the presence or absence (and concentrations) of various elements. Each characteristic x-ray line is measured in sequence by the instrument through control of the instrument geometry. The orientation of the detector crystal with respect to the sample and the photon detector is controlled synchronously such that characteristic x-ray lines can be accurately measured. The sequential measurement consists of positioning the diffraction crystal at a given theta (diffraction angle) and the detector at two-theta and counting for a given period. The crystal and detector are then rotated to a different angle for the next characteristic x-ray line.

### Neutron Activation Analysis<sup>10</sup>

*Bulk composition analysis based on gamma ray emissions induced by neutron bombardment. Typical LDLs range from parts per million to parts per billion and are dependent on the individual elements present in the sample matrix (due to decay interferences). Method is independent of electronic structure and indicative of the structure of the sample's nuclei. "For many elements and applications, NAA offers sensitivities that are superior to those attainable by other methods, on the order of parts per billion or better. In addition, because of its accuracy and reliability, NAA is generally recognized as the "referee method" of choice when new procedures are being developed or when other methods yield results that do not agree"<sup>11</sup>.*

- Radioactive isotopes created by neutron bombardment (in nuclear reactor)*
- >50 elements have stable radioactive isotopes with one neutron more than their stable isotope*

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<sup>10</sup> General Activation Analysis, Inc., "Neutron Activation Analysis: Method Description", <http://generalactivation.com/method.html>

<sup>11</sup> Michael D. Glascock, Ph.D., University of Missouri Research Reactor (MURR); "An Overview of Neutron Activation Analysis." September 12, 2005. [http://www.missouri.edu/~glascock/naa\\_over.htm](http://www.missouri.edu/~glascock/naa_over.htm)

- *By detecting the decay of these radioactive nuclei, one measures the concentration of the stable element*

The sample is introduced into the intense radiation field of a nuclear reactor where it is bombarded with neutrons, causing the elements to form radioactive isotopes. The radioactive emissions and radioactive decay paths for each element are well known. Using this information it is possible to study spectra of the emissions of the radioactive sample to determine the concentrations of the sample elements.

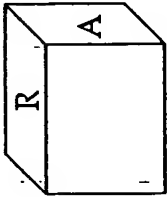
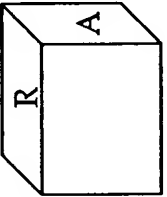
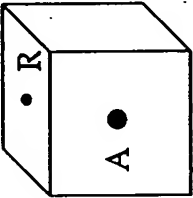
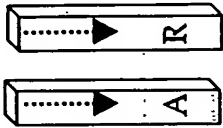
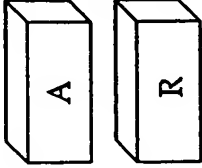


## Attachments:

Table 1: Analytical Method Overview

Table 2: Analytical Technique Overview by Activation and Detection Mechanism

Table 1: Analytical Method Overview

Technique	Penetration Depth	Sample Size	Sampled Sides	Sample Configuration	Surface Prep	Elements Tested	Activation Method	Detection Method	Typical LDL
<b>XRF</b>	1-3 mm	1" x 1" cube	axial and radial		Polished	Na – U	X-ray	X-ray (wavelength)	50 ppm
<b>PIXE</b>	40µm – 80µm	1" x 1" cube	axial and radial		Polished	Na – U	H <sup>+</sup>	X-ray (wavelength)	Varies by element (OOM ≥ppm)
<b>GD-OES</b>	0.05µm – 150µm	$\frac{1}{8}$ " diameter	axial and radial		Polished	60	n <sup>+</sup> (Ar <sup>+</sup> )	Photon (wavelength)	5 ppm
<b>GD-MS</b>	~10 mm consumed of 20mm needle	2mm x 2mm x 20 mm	axial and radial		Polished (ends only)	O – U	n <sup>+</sup> (Ar <sup>+</sup> )	Mass	Varies by element (OOM ppb)
<b>NAA</b>	Volumetric	$\frac{1}{2}$ " x 1" cube	axial and radial		Saw Cut	Na – U	Neutrons	Gamma rays (wavelength)	Varies by element (ppm-ppb)

**Table 2: Analytical Technique Overview by Activation and Detection Mechanism**

Technique	XRF	PIXE	GD-OES	GD-MS	Neutron
<b>Excited State</b>	X-ray Inner Shell e- removal	Proton Inner Shell e- removal	Plasma Ionization Ionized sputtered individual atoms	Plasma Ionization Ionized sputtered individual atoms	Neutron Bombardment Isotope creation through nucleus bombardment
<b>Detected Activity</b>	Higher energy electron moves to lower energy shell	Higher energy electron moves to lower energy shell	Ionized atoms emit energy (light) as e- move to lower energy states	Ionized atoms are separated based on weight; most weight in nucleus	Unstable isotopes decay emitting gamma rays
<b>Detection Mechanism</b>	X-ray emitted as e- moves characteristic of element	X-ray emitted as e- moves characteristic of element	Spectral lines characteristic of elements	Mass to charge ratio characteristic of elements	$1/2$ life (decay rate) characteristic of element

*Electron-based method*

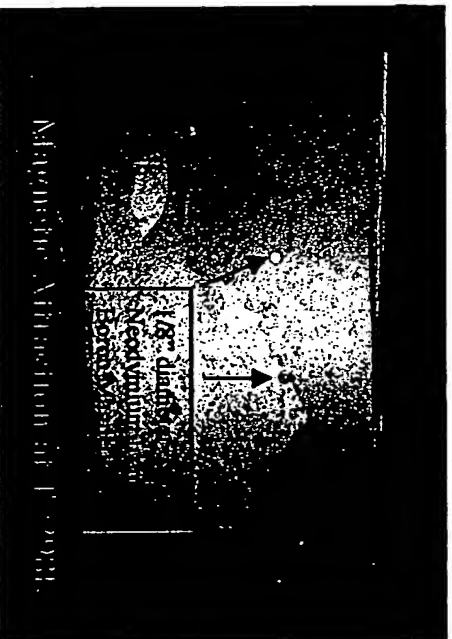
*Nucleus-based method*

## **EXHIBIT 2**

### **Photographs**

# Tailored Cu Exhibits Unique Magnetic Behavior

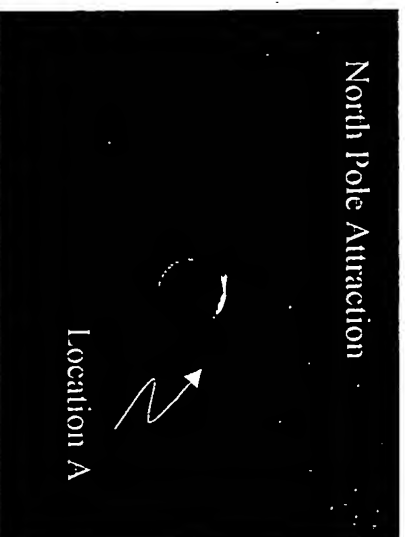
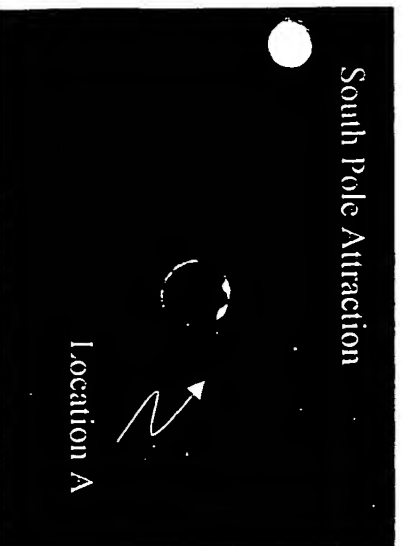
## Anisotropic Magnetic Behavior



## Tailored Cu is Magnetic:

- Anisotropic behavior: 1/8" Nd/B/Fe magnet attracted at discrete locations on radial and axial surfaces
- Radial surface exhibited greatest attraction; "Top" axial surface had greater activity than "Bottom" axial surface.
- Magnetic attraction independent of pole.

## Equivalent Attraction to North and South Pole



Ingot 14-00-01

# Elemental Copper Tailored To Reveal Magnetism



View 1

View 2

View 3

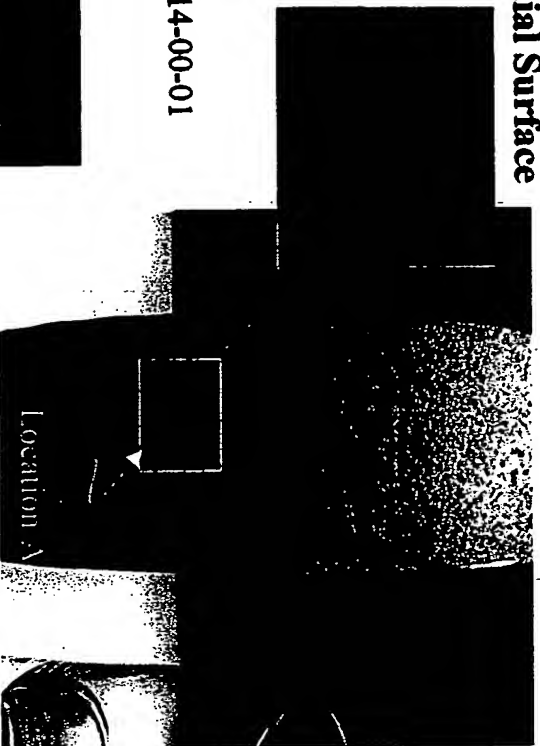
View 4

360° rotation of ingot shown

- Discrete evenly spaced points, forming a sinusoidal pattern, exhibit magnetism.
- Magnetic points exhibit equivalent attraction to the positive and negative poles of a neodymium iron boron magnet independent of temperatures below 373K.
- Magnetic point attraction to iron filings (i.e., ferromagnetism) increases linearly with decreasing temperature (e.g., 298K to 77K).
- The magnetic spots behave in accordance with known magnetic susceptibility relationships as temperature decreases (Feynman).

# Magnetic Tailored Copper Attracts Iron

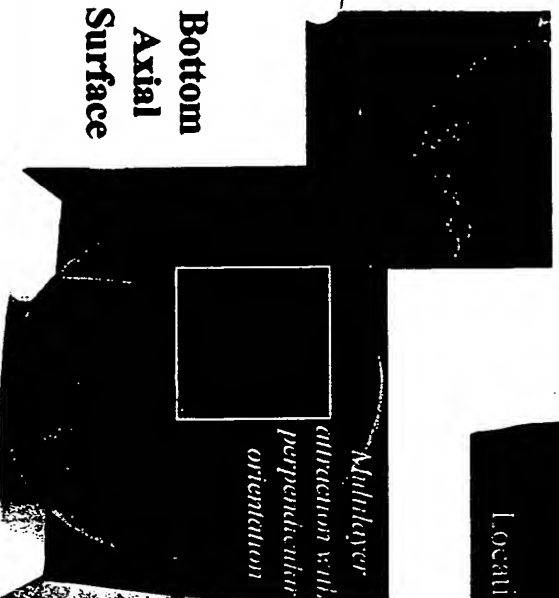
Radial Surface



Ingot 14-00-01

- Radial and bottom axial surfaces exhibited greatest activity
  - Multi-layered: strong attraction covering significant distance
  - Oriented: alignment of filings perpendicular to the surface indicative of magnetic origin of attraction
- Top axial surface showed limited regions of anisotropic magnetic behavior

Bottom  
Axial  
Surface

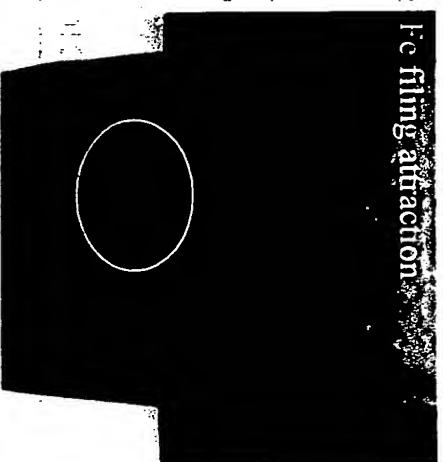
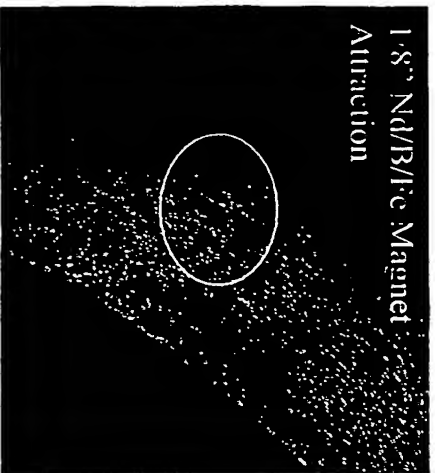


Top  
Axial  
Surface



The face of the ingot was covered with Fe filings in a horizontal orientation. The retention of Fe filings in the vertical orientation then showed areas of magnetic activity

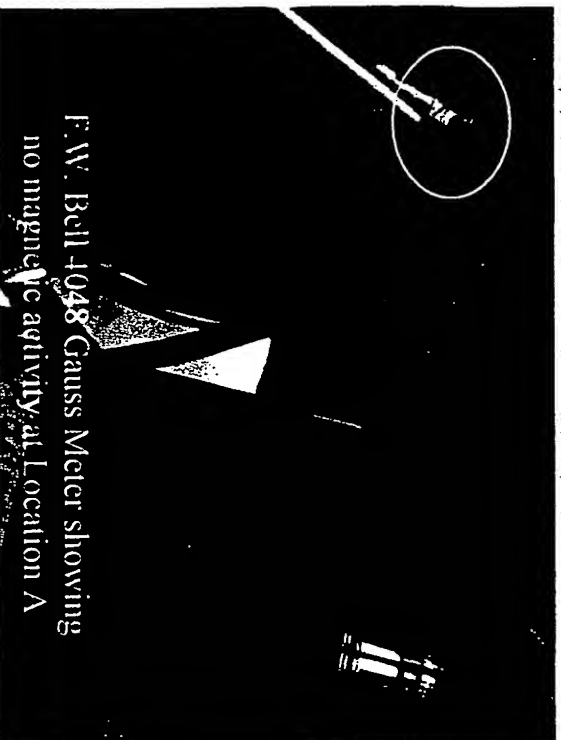
# Tailored Cu Composite Magnetic Activity



## Location A (yellow)

- Attraction to 1/8" Nd/Fe/B magnet
- Attraction to Fe filings
  - Multilayer
  - Perpendicular alignment
- No detectable Gauss reading (0.0)

Ingot  
14-00-01

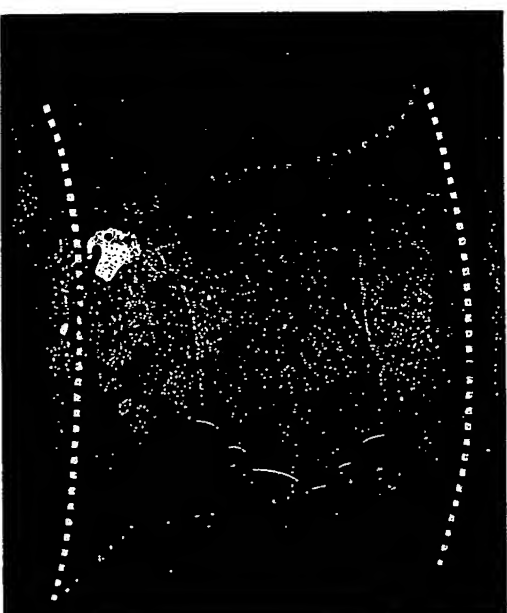
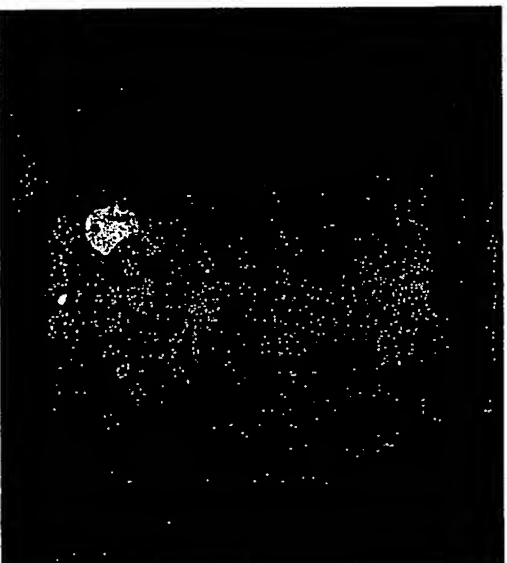
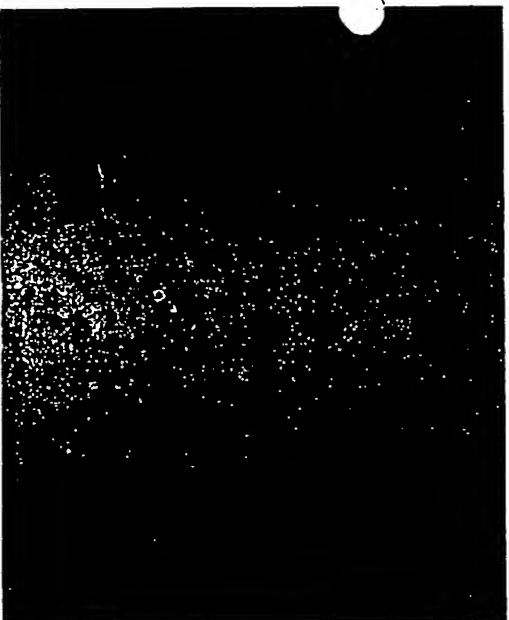


*The magnetic phenomena observed in the tailored copper is inherent to the material and unique to the tailoring/manufacturing process*



# Recursive Fractal Construction

Points depict attraction to a 1/8" diameter Nd/Fe/B magnet

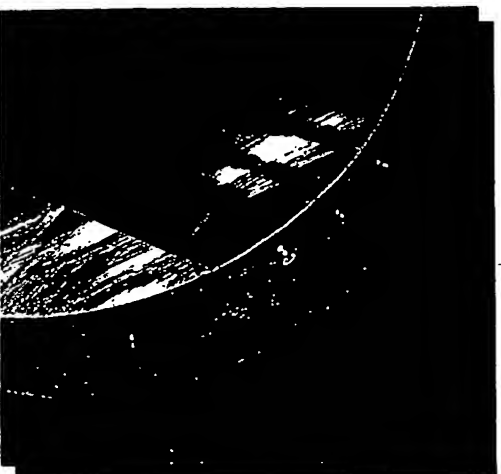


Key:

0<sup>th</sup> Order Harmonic Scaffolding    - - - -    2<sup>nd</sup> Order Harmonic Scaffolding  
.....    1<sup>st</sup> Order Harmonic Scaffolding    \_\_\_\_\_     $n^{th}$  Order Harmonic Scaffolding

- 1<sup>st</sup> order harmonic scaffolding exhibits periodicity repeating every 6 inches
- All orders of the harmonic scaffolding are constructed recursively
- The sample contains on order 550 detectable magnetic points

# Magnetic Copper: 14-00-01



Starting material:  $\geq 99.995\%_{\text{wt}}$  Cu

Comparison of Third-Party Axial and Radial Elemental Analyses via Weight-Based (GD-MS) Analytical Technique and Electronic-Structure Based (XRF) Analytical Technique (ppm)

	Al	Si	S	Cl	Ti	Fe	Cu	Rh	La	Yb	Re	Ir
GD-MS (Ax)			24				Matrix					
GD-MS (Rad)			24				Matrix					
XRF-UWO (Ax)	1350	6530		112		53	991100	372			126	118
XRF-UWO (Rad)		5640		162	53		993300	315	105	149	155	78

Blanks and all other elements non-detectable to 20 ppm

- LDLs typically  $<5$  ppm for GD-MS,  $<20$ -50 ppm for XRF
- XRF: Multiple elements (X-ray emissions) detected in radial orientation, non-detectable in axial; Fe detected only on axial XRF scan at 53 ppm
- Radial magnetic attraction noted
- High solubility of these elements in a well-mixed molten Cu bath precludes orientation-dependent concentration gradients

# Goodfellow Cambridge Limited: Magnetic Copper

## Magnetic Copper Wire

Composition:	99.96% <sub>w</sub> Cu
Wire Diameter:	0.04% <sub>w</sub> Fe
Filament Diameter:	0.5mm
Nos. of Filaments:	0.1 microns
Length:	16
Net weight:	5m
	9.1 g

## Homogeneous alloy 99.96%<sub>w</sub> Cu, 0.04%<sub>w</sub> Fe



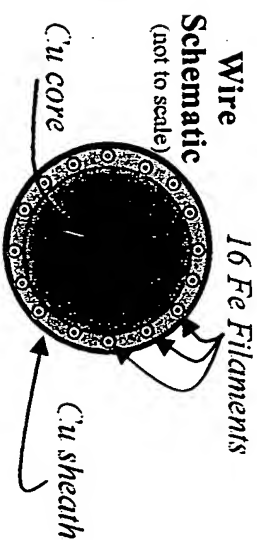
Exhibited no magnetism  
with Fe/Nd/B supermagnet



## Varying levels of compression

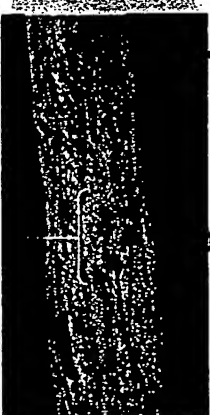


Starting  
Material

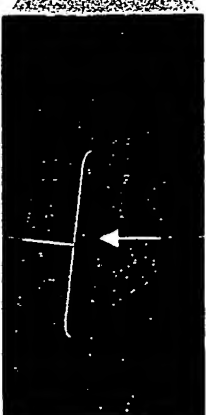


## Optical Magnification

Fe filaments

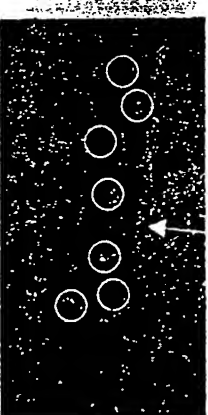


30X



30X

Fe particles after  
compression



70X

Optical spectroscopy validates that magnetism in  
Goodfellow "magnetic copper" is caused by iron  
impregnated in copper

# Natural Aluminum Manufacturer's Specification



## Certificate of Analysis

Item Number	Description	Lot Number
A-2001	ALUMINUM METAL, Al Typically 99.99% pure 2-12 mm granules	X26242

### SPECIFIC ANALYSIS OR PROPERTY

Test For	Found	Theoretical
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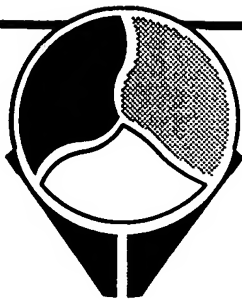
### SPECTROGRAPHIC ANALYSIS

Element	Result (%)	Element	Result (%)	Element	Result (%)
Cr	0.001	Mg	0.001		
Cu	0.002	Si	0.002		

### X-RAY DIFFRACTION ANALYSIS

CERAC Incorporated  
P.O. Box 1178, Milwaukee, WI 53201  
(414) 289-9800 Phone (414) 289-9805 Fax

*Brian T. Wegner*  
Brian T. Wegner, Q.A. Supervisor  
January 7, 2002



# OMG AMERICAS

2601 Weck Drive PO Box 12166 Research Triangle Park, NC 27709-2166

## LAB ANALYSIS FOR: QUANTUM CATALYTICS

FALL RIVER

MA 02720

SHIPPING DOC# 0406898

DATE 07/03/01

PURCHASE ORDER# 905451

CUSTOMER CODE#

GRADE: A-101-A CHIPS/E I MELT STOCK

R05

LOT NUMBER: 04106001 QUANTITY: 100 LB/ 45.36 KG HEAT NUMBER:

### CHEMICAL PROPERTIES

Carbon %	.0022
Manganese %	.0012
Nickel %	.0033
Phosphorus %	.0017
Sulfur %	.0024
Silicon %	.0010
Iron %	BALANCE

### PHYSICAL PROPERTIES

### PERFORMANCE PROPERTIES

### SPECIAL PROPERTIES

WE HEREBY CERTIFY THAT THIS MATERIAL WAS MANUFACTURED TO THE REPORTED CHEMISTRY BY OMG AMERICAS ANALYTICAL PROCEDURES FOR A-101-A CHIPS/E.I. MELT STOCK.

### COMMENTS



APPROVED: John Flynn  
TITLE: Lab Manager  
LOCATION: Johnstown Pa  
(814) 533-7800 ext 802

*John Flynn*

## **EXPERIMENTAL PROCEDURE FOR IRON 15-01-02**

### **EXAMPLE 8**

A cylindrical alumina-based crucible (99.68%  $\text{Al}_2\text{O}_3$ , 0.07%  $\text{SiO}_2$ , 0.08%  $\text{Fe}_2\text{O}_3$ , 0.04%  $\text{CaO}$ , 0.12%  $\text{Na}_2\text{O}_3$ ; 4.5" O.D. x 3.75" I.D. x 10" depth) of a 100 pound induction furnace reactor supplied by Inductotherm, fitted with a 75-30R Powertrak power supply was charged with 2000 g Iron (99.98% purity) and 200 g carbon through its charging port. The reactor was fitted with a graphite cap with a ceramic liner (i.e. the crucible, from Engineering Ceramics). During the entire procedure, a slight positive pressure of nitrogen (~0.5 psi) was maintained in the reactor using a continuous backspace purge. The reactor was heated to the metal charge liquidus point plus 300°F, at a rate no greater than 300°F/hour, as limited by the integrity of the crucible. The induction furnace operated in the frequency range of 0 kHz to 3000 kHz, with frequency determined by a temperature-controlled feedback loop implementing an Omega Model CN300 temperature controller. Upon reaching 2800°F, the reactor was charged with an additional 2595 g iron over an hour.

The temperature was again increased to 2850°F again using a rate no greater than 300°F/hour. When this temperature was reached, graphite saturation assemblies (3/8" OD, 36" long high purity (<5 ppm impurities) graphite rods) were inserted to the bottom of the iron charge through ports located in the top plate. The iron was held at 2850°F for 4 hours. Every 30 minutes during the hold period, an attempt was made to lower the graphite saturation assemblies as dissolution occurred. As the iron became saturated with carbon, the graphite saturation assemblies were consumed. After the 4 hour hold period was complete, the graphite saturation assemblies were removed.

The reactor temperature was increased to 3360°F over 7 minutes. The temperature was then varied between 2993°F and 3360°F for 15 cycles. Each cycle consisted of lowering the temperature continuously over 7 minutes and raising the temperature continuously over 7 minutes. After the 15 cycles were completed, a gas addition lance was lowered into the molten metal to a position approximately 2" from the bottom of the reactor and a 0.15 L/min flow of argon was begun. The temperature of the iron was varied over another 5 cycles between 2993°F and 3360°F.

After the fifth cycle, the reactor temperature was lowered to 2850°F over a 10 minute period with continued argon addition. The graphite saturation assemblies were reinstalled in the iron and remained there for 1 hour. The graphite saturation assemblies were removed.

The reactor temperature was lowered to 2819°F over 5 minutes. The reactor was held at this temperature for 5 minutes with continued argon addition. The temperature was then varied between 2622°F and 2818°F over 20 cycles. Each cycle consisted of lowering the temperature continuously over 9 minutes and raising the temperature continuously over 9 minutes. The argon addition ceased after completion of the 20 cycles.

The reactor temperature was lowered to 2724°F over 5 minutes. The temperature was varied between 2622°F and 2724°F over 4 ½ cycles. Each cycle consisted of lowering the temperature continuously over 5 minutes and raising the temperature continuously over 3 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added.

The reactor temperature was lowered to 2586°F over 5 minutes. The temperature was varied between 2133°F and 2586°F for 15.5 cycles. Each cycle consisted of lowering the temperature continuously over 15 minutes and raising the temperature continuously over 15 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added. All gas addition, except for the purge of nitrogen ceased after the 15.5 cycles were completed.

The temperature was varied between 2340°F and 2133°F for one cycle. The cycle consisted of raising the temperature continuously over 15 minutes and lowering the temperature continuously over 15 minutes. The gas addition lance was removed.

The reactor temperature was cooled by lowering the induction furnace power to 1 kW or less as the ingot cooled. After solidification, the iron was cooled to approximately ambient temperature in water.

## ANALYTICAL PROTOCOLS

XRF, grain size, magnetism, and chemical reactivity measurements were carried out by the procedures described in Example 1.

## ANALYTICAL RESULTS

An x-ray fluorescence analysis of the iron sample is provided in Figures 49A and 49B, with the  $K_{\alpha}$  and  $L_{\alpha}$  peaks of an iron control standard shown for reference.

An x-ray fluorescence analysis of the iron sample is provided in Figure 50A, with the  $K_{\alpha}$  peak of an aluminum control standard shown for reference.

An x-ray fluorescence analysis of the iron sample is provided in Figure 50B, with the  $K_{\alpha}$  peak of an zirconium control standard shown for reference.

An x-ray fluorescence analysis of the iron sample is provided in Figure 51A, with the  $K_{\alpha}$  peak of a sulfur control standard shown for reference.

An x-ray fluorescence analysis of the iron sample is provided in Figure 51B, with the  $K_{\alpha}$  peak of a chlorine (from potassium chloride) control standard shown for reference.

Summary data showing the apparent elemental composition of the product of Example 8 is shown in Tables 28-29, as was measured by an XRF analysis using a Uniquant software package. The apparent elemental composition of the product varies by position, which is indicated in each table.

The manufactured iron exhibited no unexpected magnetic activity. The reactivity relative to that which would be expected from natural iron has not been quantified. The ingot appears glassy or shiny on its axial (top) face and dull on its radial (side) face. The manufactured iron retained a negligible amount of refractory upon removal from the reactor, but cracked upon retrieval. The ingot had no internal voids.



Item Number	Description	Lot Number
N-1022	NICKEL METAL, Ni Typically 99.9% pure -80, +200 mesh	X0027674

**SPECIFIC ANALYSIS OR PROPERTY**

Test For	Found	Theoretical
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**SPECTROGRAPHIC ANALYSIS**

Element	Result (%)	Element	Result (%)	Element	Result (%)
Co	0.02				

**X-RAY DIFFRACTION ANALYSIS**

## **EXPERIMENTAL PROCEDURE FOR NICKEL RUN 14-01-04**

### **EXAMPLE 2**

A cylindrical alumina-based crucible (99.68%  $\text{Al}_2\text{O}_3$ , 0.07%  $\text{SiO}_2$ , 0.08%  $\text{Fe}_2\text{O}_3$ , 0.04%  $\text{CaO}$ , 0.12%  $\text{Na}_2\text{O}_3$ ; 4.5" O.D. x 3.75" I.D. x 10" depth) of a 100 pound induction furnace reactor supplied by Inductotherm, was fitted with a 75-30R Powertrak power supply and charged with 2500 g nickel (99.97% purity) and 100 g of graphite carbon through its charging port. The reactor was fitted with a graphite cap with a ceramic liner (i.e. the crucible, from Engineering Ceramics). During the entire procedure, a slight positive pressure of nitrogen (~0.5 psi) was maintained in the reactor using a continuous backspace purge. The reactor was heated to the metal charge liquidus point, over a rate no greater than 300°F/hour, as limited by the integrity of the crucible. The induction furnace operated in a frequency range of 0 kHz to 3000 kHz, with frequency determined by a temperature-controlled feedback loop implementing an Omega Model CA 300 temperature controller. Upon reaching 2800°F, the reactor was charged with an additional 2700 g nickel over an hour.

The temperature was again increased to 2850°F again using a rate no greater than 300°F/hour. When this temperature was reached, graphite saturation assemblies (3/8" OD, 36" long high purity [ $<5$  ppm impurities] graphite rods) were inserted to the bottom of the nickel charge through ports located in the top plate. The nickel was held at 2850°F for 4 hours. Every 30 minutes during the hold period, an attempt was made to lower the graphite saturation assemblies as dissolution occurred. As the nickel became saturated with carbon, the graphite saturation assemblies were consumed. After the 4 hour hold period was complete, the graphite saturation assemblies were removed.

The reactor temperature was increased to 3256°F over 7 minutes. The temperature was then varied between 2950°F and 3256°F for 15 cycles. Each cycle consisted of lowering the temperature continuously over 7 minutes and raising the temperature continuously over 7 minutes. After the 15 cycles were completed, a gas addition lance was lowered and a 0.15 L/min flow of argon was begun. The temperature of the nickel was varied over another 5 cycles between 2950°F and 3256°F.

After the fifth cycle, the reactor temperature was lowered to 2850°F over a 10 minute period with continued argon addition. The graphite saturation assemblies were reinstalled in the nickel and remained there for 1 hour. The graphite saturation assemblies were removed.

The reactor temperature was lowered to 2829°F over 5 minutes. The reactor was held at this temperature for 5 minutes with continued argon addition. The temperature was then varied between 2790°F and 2829°F over 20 cycles. Each cycle consisted of lowering the temperature continuously over 9 minutes and raising the temperature continuously over 9 minutes. The argon addition ceased after completion of the 20 cycles.

The reactor temperature was lowered to 2770°F over 5 minutes. The temperature was varied between 2710°F and 2770°F over 4 ½ cycles. Each cycle consisted of lowering the temperature continuously over 5 minutes and raising the temperature continuously over 3 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added.

The reactor temperature was lowered to 2691°F over 5 minutes. The temperature was varied between 2492°F and 2691°F for 15.5 cycles. Each cycle consisted of lowering the temperature continuously over 15 minutes and raising the temperature continuously over 15 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added. All gas addition, except for the purge of nitrogen ceased after the 15.5 cycles were completed.

The temperature was varied between 2571°F and 2492°F for one cycle. The cycle consisted of raising the temperature continuously over 15 minutes and lowering the temperature over 15 minutes. The gas addition lance was removed.

The reactor temperature was slowly cooled by lowering the induction furnace power to 1 KW or less as the ingot cooled. The nickel was then cooled to approximately ambient temperature in water.

## ANALYTICAL PROTOCOLS

XRF, grain size, magnetism, and chemical reactivity measurements were carried out by the procedures described in Example 1.

## ANALYTICAL RESULTS

An x-ray fluorescence analysis of the nickel sample is provided in Figures 28A and 28B, with the  $K_{\alpha}$  and  $L_{\alpha}$  peaks of a nickel control standard shown for reference.

An x-ray fluorescence analysis of the nickel sample is provided in Figure 29A, with the  $K_{\alpha}$  peak of an aluminum control standard shown for reference.

An x-ray fluorescence analysis of the nickel sample is provided in Figure 29B, with the  $K_{\alpha}$  peak of a zirconium control standard shown for reference.

An x-ray fluorescence analysis of the nickel sample is provided in Figure 30A, with the  $K_{\alpha}$  peak of a sulfur control standard shown for reference.

An x-ray fluorescence analysis of the copper sample is provided in Figure 30B, with the  $K_{\alpha}$  peak of an chlorine (from potassium chloride) shown for reference.

Summary data showing the apparent elemental composition of the product of Example 2 is shown in Tables 14-16, as was measured by an XRF analysis using a Uniquant software package. The apparent elemental composition of the product varies by position, which is indicated in each table.

The manufactured nickel retained a large amount of refractory on its exterior surface after retrieval from the reactor. The retained refractory was attributed to either surface attraction or reaction with the high content of  $Al_2O_3$  in the refractory. The ingot did not crack with handling, but did have an internal void. The visible radial surface appeared duller in than the axial (top) face, again demonstrating anisotropic physical properties. The ingot demonstrated no unexpected chemical reactivity after removal from the reaction system.

Item Number	Description	Lot Number
C-1226	COBALT METAL, Co Typically 99.5% pure (excluding Ni) 12 mm shot & smaller	X0030613

## SPECIFIC ANALYSIS OR PROPERTY

Test For	Found	Theoretical
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## SPECTROGRAPHIC ANALYSIS

Element	Found (%)	Element	Found (%)	Element	Found (%)
Fe	0.21	Ni	0.265		

## X-RAY DIFFRACTION ANALYSIS



PO Box 1178  
MILWAUKEE, WI 53201

Telephone : 414-289-9800  
Fax : 414-289-9805

## Sales Packing slip

Customer Acct # .....: CUS-071  
Number .....: PAC-016366  
Date .....: 12/22/04  
Page .....: 1  
Sales order .....: SO-264965  
Purchase order .....: 20904  
Your ref. ....:  
Mode of delivery .....: UPS Ground  
Terms of delivery .....: FOB Origin  
Freight code .....: PrePay & Add  
Contact Name .....: Janet Pichette  
Contact Phone .....: 508-324-6411  
Contact Fax .....:  
Contact Email .....

Sold to : Atomic Ordered Materials, LLC  
22 Evergreen Dr.  
SEEKONK, MA 02771

Ship to : Atomic Ordered Materials, LLC  
421 Currant Road  
FALL RIVER, MA 02720

\*\*\*\*\* Header Comments \*\*\*\*\*

mk

Item number	Name	Ordered	Delivered	Balance
C-1226	COBALT METAL Co 12 mm shot & smaller Typ. 99.5% (ex Ni) 7440-48-4 Country of Origin: ZM COBALT METAL	20,000.00	20,000.00	

Quantity : 20,000.00 Warehouse : 1 Batch number : X0030613 Location : 000-0-0  
Per Quote 11/04

Schedule B# 8105.20.0000  
ECCN # EAR99

Receipt: \_\_\_\_\_

## **EXPERIMENTAL PROCEDURE FOR COBALT RUN 14-01-05**

### **EXAMPLE 3**

A cylindrical alumina-based crucible (99.68%  $\text{Al}_2\text{O}_3$ , 0.07%  $\text{SiO}_2$ , 0.08%  $\text{Fe}_2\text{O}_3$ , 0.04%  $\text{CaO}$ , 0.12%  $\text{Na}_2\text{O}_3$ ; 4.5" O.D. x 3.75" I.D. x 10" depth) of a 100 pound induction furnace reactor supplied by Inductotherm and fitted with a 75-30R Powertrak power supply, was charged with 2176 g cobalt (99.8% purity) through its charging port. The reactor was fitted with a graphite cap with a ceramic liner from Engineering Ceramics. During the entire procedure, a slight positive pressure of nitrogen (~0.5 psi) was maintained in the reactor using a continuous backspace purge. The reactor was heated to 2800°F over a minimum of 14 hours while the induction furnace operated in a frequency range of 0 kHz to 3000 kHz. Upon reaching 2700°F, the reactor was charged with an additional 3000 g cobalt over an hour.

When 2800°F was reached, graphite saturation assemblies were inserted to the bottom of the cobalt charge through ports located in the top plate. The cobalt was held at 2800°F for 4 hours. Every 30 minutes during the hold period, an attempt was made to lower the graphite saturation assemblies as dissolution progressed. As the cobalt became saturated with carbon, the graphite saturation assemblies were consumed. After the 4 hour hold period was complete, the graphite saturation assemblies were removed.

The reactor temperature was increased to 3086°F over 7 minutes. The temperature was then varied between 2875°F and 3086°F for 15 cycles. Each cycle consisted of lowering the temperature continuously over 7 minutes and raising the temperature continuously over 7 minutes. After the 15 cycles were completed, a gas addition lance was lowered and a 0.15 L/min flow of argon was begun. The temperature of the cobalt was varied over another 5 cycles between 2875°F and 3086°F.

After the fifth cycle, the reactor temperature was lowered to 2800°F over a 10 minute period with continued argon addition. The graphite saturation assemblies were reinstalled in the cobalt and remained there for 1 hour. The graphite saturation assemblies were removed.

The reactor temperature was lowered to 2785°F over 5 minutes. The reactor was held at this temperature for 5 minutes with continued argon addition. The temperature

was then varied between 2689°F and 2785°F over 20 cycles. Each cycle consisted of lowering the temperature continuously over 9 minutes and raising the temperature continuously over 9 minutes. The argon addition ceased after completion of the 20 cycles.

The reactor temperature was lowered to 2737°F over 5 minutes. The temperature was varied between 2689°F and 2737°F over 4 ½ cycles. Each cycle consisted of lowering the temperature continuously over 5 minutes and raising the temperature continuously over 3 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added.

The reactor temperature was lowered to 2672°F over 5 minutes. The temperature was varied between 2498°F and 2672°F for 15.5 cycles. Each cycle consisted of lowering the temperature continuously over 15 minutes and raising the temperature continuously over 15 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added. All gas addition, except for the purge of nitrogen ceased after the 15.5 cycles were completed.

The temperature was varied between 2570°F and 2498°F for one cycle. The cycle consisted of raising the temperature continuously over 15 minutes and lowering the temperature continuously over 15 minutes. The gas addition lance was removed.

The reactor temperature was cooled by lowering the induction furnace power to 1 kW or less as the ingot cooled. The cobalt was then cooled to approximately ambient temperature in water.

## ANALYTICAL PROTOCOLS

XRF, grain size, magnetism, and chemical reactivity measurements were carried out by the procedures described in Example 1.



## ANALYTICAL RESULTS

An x-ray fluorescence analysis of the cobalt sample is provided in Figures 31A and 31B, with the  $K_{\alpha}$  and  $L_{\alpha}$  peaks of a cobalt control standard shown for reference.

An x-ray fluorescence analysis of the cobalt sample is provided in Figure 32A, with the  $K_{\alpha}$  peak of an aluminum control standard shown for reference.

An x-ray fluorescence analysis of the cobalt sample is provided in Figure 32B, with the  $K_{\alpha}$  peak of an iron control standard shown for reference.

An x-ray fluorescence analysis of the cobalt sample is provided in Figure 33A, with the  $K_{\alpha}$  peak of a chlorine (from potassium chloride) control standard shown for reference.

An x-ray fluorescence analysis of the cobalt sample is provided in Figure 33B, with the  $K_{\alpha}$  peak of a zirconium control standard shown for reference.

An x-ray fluorescence analysis of the cobalt sample is provided in Figure 34, with the  $K_{\alpha}$  peak of a manganese control standard shown for reference.

Summary data showing the apparent elemental composition of the product of Example 3 is shown in Table 17, as was measured by an XRF analysis using a Uniquant software package.

The top (axial) face of the manufactured cobalt ingot exhibited many of the recursive patterns observed in other manufactured ingots. The surface peaks are inconsistent with what would be expected given the forces of gravity during cooling. In addition, the shiny top face of the ingot exhibited an unexpected coloration, such that some of the faces had a distinct pink tint. While the top of the ingot was shiny, silver metallic, the sides of the ingot were matte silver in appearance.

The manufactured cobalt ingot retained a small amount of refractory around its base. The ingot did not crack upon retrieval from the reaction system. No unexpected magnetic behavior or chemical reactivity were observed.

Compare #1		Drop at 1" data	11/15/2005	10/24/2005	11/21/2005	1/9/2006	1/9/2006
		GDOES	GDOES	XRF-JWO	POE	GDOES	GDOES
		Al chunks	Aluminum	Aluminum	Aluminum	5 ppm OEM Limit	5 ppm OEM Limit
		Average	14-01-10	14-01-10	14-01-10	Aluminum	Aluminum
		Max	Al B (90.9%wt)	Al B (90.9%wt)	Al B (90.9%wt)	Al B (90.9%wt)	Al B (90.9%wt)
		99.9999	total	total	total	40-45	40-45
			radial	radial	radial	Ave 0.05-15 Ave 15-100	Ave 0.05-15 Ave 15-130
1	H						
2	He						
3	Li						
4	Be						
5	B	0.05	#DIV/0!			118	108.9
6	C			23		1812	501.4
7	N					364.2	101.7
8	O		110	220	180540	434010	7418
9	F					32100	7869
10	Ne					6222	12280
11	Na	0.17800007	0.2442188				8351
12	Mg	14.33333333	17.7109296				
13	Al			470			
14	Si	21.80000007	28.4218597	886300	981900	637250	558740
15	P	0.498000007	0.64382269	9400	31800	983400	850400
16	S	0.288000007	0.4354649			982800	863400
17	Cl	0.075	0.20165067			850400	828800
18	Ar			253	811	815.2	1341
19	K					22.78	57.49
20	Ca	0.5	#DIV/0!			787.097	103.1
21	Sc	0.029000007	0.04355405			305.9	107.8
22	Ti	0.41	0.88885095			98.82	287
23	V	0.1833333333	0.23209014			1100	101.2
24	Cr	0.298000007	0.44382269				
25	Mn	0.1033333333	0.1371082				
26	Fe	14.33333333	17.7109296	770	790		
27	Co	0.058000007	0.17488255	2230	1950		
28	Ni	0.2833333333	0.83704287			1270	1720
29	Cu	13.90000007	17.042832			1720	1117
30	Zn	1.900000007	3.1647842	34	30	48.222	440.3
31	Ga	0.298000007	0.44382269	81	79	1987	1081
32	Ge			387	583	440.3	444.1
33	As					1228	1500
34	Se					21.729	32.878
35	Br					84.458	58.375
36	Kr						
37	Rb						
38	Sr						
39	Y	0.025	0.05033197				
40	Zr	0.1233333333	0.21269014				
41	Nb						
42	Mo	0.0633333333	0.0871093				
43	Tc						
44	Ru						
45	Rh						
46	Pd						
47	Ag						
48	Cd						
49	In						
50	Sn						
51	Sb						
52	Te						
53	I						
54	Xe						
55	Cs						
56	Ba	0.150000007	0.45687401				
57	La	0.33	1.00487438				
58	Ce	0.4233333333	1.33403258				
59	Pr	0.118000007	0.35308842				
60	Nd	0.3733333333	1.17580016				
61	Pm						
62	Sm						
63	Eu						
64	Gd	0.88	#DIV/0!				
65	Tb						
66	Dy						
67	Ho						
68	Er	0.02	#DIV/0!				
69	Tm						
70	Yb						
71	Lu						
72	Hf						
73	Ta						
74	W	0.298000007	0.87557184				
75	Re						
76	Os						
77	Ir						
78	Pt						
79	Au						
80	Hg						
81	Tl						
82	Pb	0.08	0.11066385				
83	Bi						
84	Po						
85	At						
86	Rn						
87	Fr						
88	Ra						
89	Ac						
90	Th	0.08	0.1813179				
91	Pa						
92	U	0.8188	0.80581163	80.10%	83.70%	13.84%	14.13%
93	Np						
94	Pu						
95	Am						
96	Cm						
97	Bk						
98	Cf						
99	Es						
100	Fm						
101	Md						
102	No						
103	Lr						
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Compare at		GDMS	10/26/2005	10/24/2005	11/21/2005	1/6/07	1/6/2008
		Confidence Interval	GDMS	XRF-JWD	PIXIE	OD	GOOE8
		Cu Chop	Copper	Copper	Copper	5 ppm OEM Limit	5 ppm OEM Limit
		Average	Patent	Patent	Patent	Copper	Copper
		Max	14-00-01	14-00-01	14-00-01	14-00-01A	14-00-01R
		99.9999	Patent	Patent	Patent	Patent	Patent
			total	total	total	40-45	40-45
			radial	radial	radial	Ave 0.05-15	Ave 0.05-15
						Ave 15-155	Ave 15-115
1	H						
2	He						
3	Li						
4	Be						
5	B	0.0010	0.0010				
6	C					63.25	56.12
7	N						
8	O				235.250	128.860	166.3
9	F					3290	1534
10	Ne						634.3
11	Na						3142
12	Mg	0.0040	0.0125			286.6	249
13	Al	0.3938	2.3018	1350	958.198	314	249
14	Si	0.0760	0.1689	6530	1910	50.15	126.6
15	P	0.0130	0.0485		1400	255.6	2158
16	S	3.8600	8.3912		6320	1823	2158
17	Cl	0.1260	0.2148	24		20.7	22.01
18	Ar			112	350.719	40.23	45.06
19	K	0.0300	0.0300	162	212.406	20.47	
20	Ca						
21	Sc					20.75	24.22
22	Ti	0.0114	0.0302				
23	V			53			
24	Cr	0.0340	0.1857				
25	Mn	0.0136	0.0381				
26	Fe	2.1520	4.8122	53	472.395	62.38	53.6
27	Co	0.0140	0.0382		461.63	73.99	61.79
28	Ni	0.5620	1.0635		56.96	56.74	37.93
29	Cu			891100	862230	52.08	40.85
30	Zn			993300	995000	993900	995500
31	Ga					996100	993500
32	Ge						995900
33	As	0.4760	0.6351				
34	Se	0.8180	0.9602				
35	Br						
36	Kr						
37	Rb						
38	Sr						
39	Y						
40	Zr						
41	Nb						
42	Mo	0.0153	0.0390				
43	Tc						
44	Ru						
45	Rh			372	315		
46	Pd						
47	Ag	8.7800	10.2431			42.7	43.75
48	Cd					40.81	42.63
49	In						43.48
50	Sn	0.2900	0.7513				42.11
51	Sb	0.4960	1.1273			113.7	141.1
52	Te	0.1340	0.3199			116.3	126.1
53	I						166.7
54	Xe					71.09	91.3
55	Cs					45.64	97.42
56	Ba	0.0035	0.0080			87.97	97.42
57	La			105			
58	Ce						
59	Pr						
60	Nd						
61	Pm						
62	Sm						
63	Eu						
64	Gd						
65	Tb						
66	Dy						
67	Ho						
68	Er						
69	Tm						
70	Yb						
71	Lu			149			
72	Hf						
73	Ta						
74	W	0.2183	1.1874				
75	Re			126	155		
76	Os						
77	Ir			118	78		
78	Pt						
79	Au						
80	Hg						
81	Tl						
82	Pb	0.7820	1.8213			81.85	83.3
83	Bi	0.4218	0.9733			83.1	109.3
84	Po					85.1	104
85	At					87.81	109.3
86	Rn					89.8	85.83
87	Fr						
88	Ra						
89	Ac						
90	Th						
91	Pa						
92	U						
				99.00%	100.00%	78.475	82.114
						200.00	110.30
						N/A	0.2

Compare at		7/17/2006		7/17/2006		7/17/2006		5/24/2001		12/10/2005	
		GDMS		GDMS		POCE		XRF-ADM		MAA	
		Confidence Interval		Silicon 14-06-05		Silicon 15-01-01		Silicon 15-01-01		Silicon 15-01-01	
		SI Powder		Starting Material		Patent					
		Average	Max	n/a							
		99.9999									

Compare at	Unit	7/10/2008	7/14/2008	7/17/2008	6/1/2001
7/1	analysis	GDMS	GDMS	PCE	XRF-ADM
7/2	analysis	Iron	Iron	Iron	Iron
7/3	analysis	Starting Material	15-01-02	15-01-02	15-01-02
7/4	analysis	n/a		Potent	
File		total	radial	total	radial
1	H				
2	He				
3	Li				
4	Be				
5	B				
6	C				
7	N				
8	O			86000	22510
9	F				
10	Ne				
11	Na				400
12	Mg				
13	Al	5100	6200	10080	20240
14	Si	480	480	1370	2340
15	P	52	31		33
16	S	37	23		281,985
17	Cl				40
18	Ar				120
19	K				130
20	Ca			183,727	53
21	Sc				25
22	Ti				73
23	V				26
24	Cr	21			
25	Mn				
26	Fe			901980	954430
27	Co		21		980100
28	Ni	44	36		967800
29	Cu				120
30	Zn				140
31	Ga				
32	Ge				
33	As				
34	Se				
35	Br				
36	Kr				
37	Rb				
38	Sr				
39	Y				
40	Zr	180	110		99
41	Nb				120
42	Mo				
43	Tc				
44	Ru				
45	Rh				
46	Pd				
47	Ag				
48	Cd				
49	In				
50	Sn				
51	Sb				
52	Te				
53	I				
54	Xe				
55	Cs				
56	Ba				
57	La				
58	Ce				
59	Pr				
60	Nd				
61	Pm				
62	Sm				
63	Eu				
64	Gd				
65	Tb				
66	Dy				
67	Ho				
68	Er				
69	Tm				
70	Yb				
71	Lu				
72	Hf				
73	Ta				
74	W				
75	Re				90
76	Os				
77	Ir				
78	Pt				
79	Au				
80	Hg				
81	Tl				
82	Pb				
83	Bi				
84	Po				
85	At				
86	Rn				
87	Fr				
88	Ra				
89	Ac				
90	Th				80
91	Pa				
92	U				
		81.34	87.749	99.80%	97.80%

Compare at	date	7/10/2006	7/17/2006	7/17/2006	6/1/2001
70	analysis	GDMS	GDMS	PDCE	XRF-ADM
70	inclusion				
70	sample type	Cobalt	Cobalt	Cobalt	Cobalt
70	sample	Starting Material	14-01-05	14-01-05	14-01-05
70	sample	info	info	info	info
			total	total	total
			radial	radial	radial
1	H				
2	He				
3	Li				
4	Be				
5	B				
6	C				
7	N				
8	O			79470	
9	F				
10	Ne				
11	Na				290
12	Mg				430
13	Al	500	5000	33280	49000
14	Si	55	650	3480	730
15	P		25		40
16	S	47	90	730.378	577.381
17	Cl			748.054	
18	Ar				47
19	K				110
20	Ca			381.507	201.664
21	Sc				75
22	Ti				22
23	V				33
24	Cr				
25	Mn	90	90		81
26	Fe		1000	1550	1300
27	Co			95830	908330
28	Ni		1500		1800
29	Cu	120	130		1800
30	Zn	50	130		
31	Ga				
32	Ge				
33	As				
34	Se				
35	Br				
36	Kr				
37	Rb				
38	Sr				
39	Y				
40	Zr		100	200	190
41	Nb			130	
42	Mo		25	50	
43	Tc				
44	Ru				
45	Rh				
46	Pd				
47	Ag				
48	Cd				30
49	In				
50	Sn				
51	Sb				
52	Te				
53	I				
54	Xe				
55	Cs				
56	Ba				80
57	La				
58	Ce				
59	Pr				
60	Nd				
61	Pm				
62	Sm				
63	Eu				80
64	Gd				100
65	Tb				110
66	Dy				
67	Ho				
68	Er				
69	Tm				
70	Yb				
71	Lu				100
72	Hf				
73	Ta				
74	W		50		
75	Re				
76	Os				
77	Ir				
78	Pt				
79	Au				
80	Hg				
81	Tl				
82	Pb				
83	Bi				
84	Po				
85	At				
86	Rn				
87	Fr				
88	Ra				
89	Ac				
90	Th				
91	Pa				
92	U				
			89.683	87.683	101.854
					101.854

Compare at		7/10/2006	7/10/2006	7/17/2006	7/17/2006	5/24/2007	5/24/2007
		GDMS	GDMS	PDE	PDE	XRF-AOM	XRF-AOM
		Nickel Starting Material n/a	Nickel 14-01-04	Nickel 14-01-04 Patent	Nickel 14-01-04	Nickel 14-01-04	Nickel 14-01-04
From	To	total	radial	total	radial	total	radial
1	H						
2	He						
3	Li						
4	Be						
5	B						
6	C						
7	N						
8	O						
9	F			80850	30420		
10	Ne						
11	Na					310	1200
12	Mg						
13	Al			17520	19470	21100	25700
14	Si	190	250	516.734	868.145	300	1400
15	P						
16	S						
17	Cl						
18	Ar						50
19	K						100
20	Ca				780.087		48
21	Sc						110
22	Ti					24	
23	V						
24	Cr						
25	Mn						
26	Fe	34	30		240.878		
27	Cs						
28	Ni			921120	948200	971000	984300
29	Cu						
30	Zn						
31	Ga						
32	Ge						50
33	As						
34	Se						
35	Br						
36	Kr						
37	Rb						
38	Sr						
39	Y						
40	Zr	240	250			370	330
41	Nb						
42	Mo						
43	Tc						
44	Ru						
45	Rh						
46	Pd					50	
47	Ag						
48	Cd						
49	In						
50	Sn						
51	Sb						
52	Te						
53	I						
54	Xe						
55	Cs						
56	Ba						
57	La						60
58	Ce						
59	Pr						
60	Nd						
61	Pm						
62	Sm						
63	Eu						
64	Gd						
65	Tb						
66	Dy						
67	Ho						
68	Er						
69	Tm						
70	Yb						
71	Lu					8900	8400
72	Hf						
73	Ta						
74	W						
75	Re						
76	Os						
77	Ir						
78	Pt						
79	Au						
80	Hg						
81	Tl						
82	Pb						
83	Bi						
84	Po						
85	At						
86	Rn						
87	Fr						
88	Ra						
89	Ac						
90	Th						
91	Pa						
92	U						
		72.813	84.821	1121.021	100.878		

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